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Sodium 3,5-dinitropyridin-4-olate dihydrate

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Key indicators

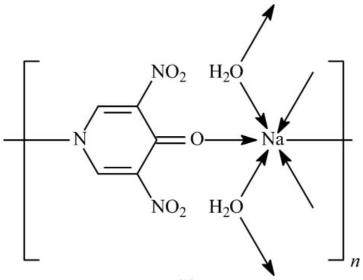
Single-crystal X-ray study
T = 295 K
Mean *σ*(C–C) = 0.002 Å
R factor = 0.044
wR factor = 0.132
Data-to-parameter ratio = 13.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The six-membered ring of the 3,5-dinitropyridin-4-olate anion in the title dihydrated sodium derivative, $Na^+\cdot C_5H_2N_3O_5^-\cdot 2H_2O$, exists in the pyridone form with C=C and C=O double bonds, and the negative charge formally residing on the N atom. The anions link the Na^+ ions, each of which is coordinated by four water molecules, into a linear chain that runs along the *c* axis of the unit cell. The chains are bridged by the water molecules into a sheet structure. The anion and the Na^+ ion lie on a mirror plane.

Comment

3,5-Dinitropyridine exists as a monomeric molecule whose aromatic ring is delocalized (Destro *et al.*, 1974); the 4-hydroxy-substituted derivative, a herbical compound (Diehl & Walworth, 1970), has long been known to exist in the pyridinone form (Crowe, 1925) although this has not yet been shown by crystal structure analysis. The substituted compound can be regarded as an analog of picric acid (Koziol & Tomasik, 1977); in its deprotonated form, the 3,5-dinitropyridin-4-olate anion features a C—O bond of a length typical of carbonyl bonds, as noted from the crystal structure of its (oxonium)tris(triphenylphosphine)trigold (Kuz'mina *et al.*, 1997, 1999) salt.



(I)

The title compound, (I) (Fig. 1), which was synthesized many years ago (Crowe, 1925), has a formal negative charge on the N atom. The six-membered ring of the 3,5-dinitropyridin-4-olate anion exists as the localized form, with C=C [1.377 (2) Å] and C=O [1.238 (3) Å] double bonds. The anions link the Na^+ ions, each of which is coordinated by four water molecules, into a linear chain that runs along the *c* axis of the unit cell. The chains are linked through the water molecules, each of which interacts with two Na^+ ions, into a layer. Hydrogen bonds (Table 2) consolidate the layer motif.

Experimental

Sodium hydroxide (0.40 g, 10 mmol) was added to 3,5-dinitropyridin-4-one (1.85 g, 10 mmol) suspended in water (20 ml). The suspension was heated to 343 K until it had dissolved completely. The solution

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was concentrated and then set aside for the growth of crystals. Yellow–orange crystals were isolated after several weeks.

Crystal data

$Na^+ \cdot C_5H_2N_3O_5^- \cdot 2H_2O$
M_r = 243.12
Monoclinic, $P2_1/m$
a = 3.6392 (5) Å
b = 14.446 (2) Å
c = 8.859 (1) Å
β = 98.876 (1)°
V = 460.2 (1) Å^3
Z = 2
D_x = 1.755 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.20 mm^{-1}
T = 295 (2) K
Block, yellow–orange
0.40 × 0.27 × 0.17 mm

Data collection

Bruker APEX2 area-detector diffractometer
φ and ω scans
Absorption correction: none
3973 measured reflections
1101 independent reflections
957 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.015
$\theta_{\rm max}$ = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.132
S = 1.10
1101 reflections
79 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0692P)^2 + 0.1823P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max}$ = 0.001
$\Delta\rho_{\rm max}$ = 0.31 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.35 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Na1—O1	2.482 (2)	Na1—O1*w*^{ii}	2.441 (2)
Na1—O1*w*	2.373 (2)	Na1—O1*w*^{iii}	2.373 (2)
Na1—O1*w*^{i}	2.441 (2)	Na1—N2^{iv}	2.479 (2)
O1—Na1—O1*w*	91.66 (6)	O1*w*—Na1—N2^{iv}	107.75 (6)
O1—Na1—O1*w*^{i}	71.27 (6)	O1*w*^{i}—Na1—O1*w*^{ii}	77.87 (7)
O1—Na1—O1*w*^{ii}	71.27 (6)	O1*w*^{i}—Na1—O1*w*^{iii}	162.89 (8)
O1—Na1—O1*w*^{iii}	91.66 (6)	O1*w*^{i}—Na1—N2^{iv}	88.94 (6)
O1—Na1—N2^{iv}	154.26 (8)	O1*w*^{ii}—Na1—O1*w*^{iii}	98.23 (5)
O1*w*—Na1—O1*w*^{i}	98.23 (5)	O1*w*^{ii}—Na1—N2^{iv}	88.94 (6)
O1*w*—Na1—O1*w*^{ii}	162.89 (8)	O1*w*^{iii}—Na1—N2^{iv}	107.75 (6)
O1*w*—Na1—O1*w*^{iii}	80.55 (7)		

Symmetry codes: (i) $x-1, y, z$; (ii) $x-1, -y+\frac{3}{2}, z$; (iii) $x, -y+\frac{3}{2}, z$; (iv) $x, y, z+1$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O1*w*—H2⋯O2^{v}	0.85	2.41	3.063 (2)	134
O1*w*—H2⋯O3^{v}	0.85	2.33	3.127 (2)	157
O1*w*—H1⋯O2^{vi}	0.85	2.17	2.922 (2)	147

Symmetry codes: (v) $-x+1, -y+1, -z+1$; (vi) $x+1, y, z$.

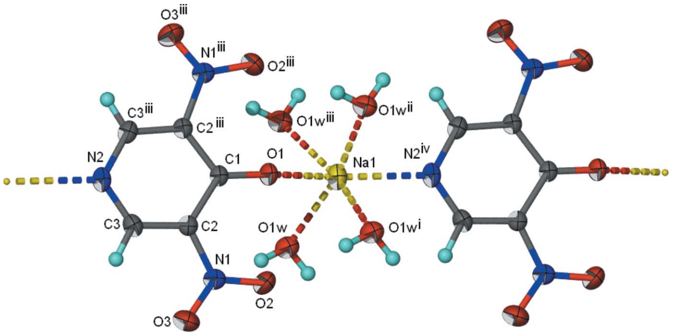

Figure 1
A portion of the chain structure of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms as spheres of arbitrary radii. Symmetry codes are given in Table 1.

H atoms were treated as riding, with C—H = 0.93Å and O—H = 0.85Å, and were included in the refinement with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C,O).

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2006).

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